Citation 2:

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Application No. 10-184035 – 30 June 1998

Applicant: MITSUBISHI CHEMICAL CORPORATION, Tokyo, JP

Title: METHOD FOR THE PRODUCTION OF QUATERNARY AMMONIUM TETRAFLUOROBORATES

[Claims]

1. A method for the production of a quaternary ammonium tetrafluoroborate by reacting a quaternary ammonium bicarbonate or carbonate monoester according to the general formula $Q^+ \cdot RCO_3^-$ wherein R is a hydrogen atom or an alkyl group having 1 to 4 carbon atoms and Q^+ is a quaternary ammonium group with hydrogen fluoride to generate a quaternary ammonium fluoride which is then reacted with boron trifluoride or a complex containing the same.

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[Excerpt of the descriptive part of the specification]

[0001] [Technical Field of the Invention]

The present invention relates to a method for the production of a quaternary ammonium tetrafluoroborate by a process involving a salt exchange. While a quaternary ammonium tetrafluoroborate obtained according to the invention is employed preferably as an electrolyte for an electrochemical element such as a cell or an electrolytic condenser as it is extremely pure, it can also be employed widely in various fields as a surfactant, an phase transfer catalyst, a softening agent, a antistatic agent such as a detergent, a dispersant for an asphalt or cement, a bactericide, a preservative, an anti-blocking agent or an anti-aggregating agent for a fertilizer or a granular material. .....

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Example 1

A 1000 mL-volume reaction vessel made of fluorocarbon resin and fitted with a stirrer was charged with 500 mL of a methanol solution containing 191.24 g (1.0 mole) triethylmethylammonium methylcarbonate ($(C_2H_5)_3(CH_3)N^+ \cdot CH_3CO_3^-$) obtained by reacting triethylamine with methyl carbonate. While keeping at 0°C, 30 mL of cooled liquid hydrogen fluoride (1.5 moles as HF) was added dropwise over a period of 1 hour while stirring. After stirring for one further hour, a methanol solution of triethylmethylammonium fluoride containing excessive hydrogen fluoride was obtained. This solution while being kept at 0°C was treated dropwise slowly with 133.0 g of a boron trifluoride methanol solution containing boron trifluoride at 51% (1.0 mole as BF_3) over a period of 30 minutes. The reaction vessel was immersed in an oil bath at 100°C to distill off a large portion of methanol and excessive hydrogen fluoride. The reaction vessel was purged with nitrogen to remove methanol and hydrogen fluoride to obtain 200 g (1.0 mole) of triethylmethylammonium tetrafluoroborate ($(C_2H_5)_3(CH_3)N^+ \cdot BF_4^-$). Chemical analysis showed a purity of the resultant triethylmethylammonium tetrafluoroborate on the basis of BF_4 of 99.5% or more, with impurities such as chloride ion or bromide ion being less than 1 ppm which is a detection limit of a nephelometry. .....

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PRODUCTION OF QUATERNARY AMMONIUM TETRAFLUOROBORATE

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Inventor(s): HASEGAWA KATSUAKI; NAGAOKA HIDEO; UE MAKOTO; TAKEHARA MASAHIRO +
Applicant(s): MITSUBISHI CHEM CORP +
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Abstract of **JP 2000016995 (A)**
PROBLEM TO BE SOLVED: To provide a method for producing a highly pure quaternary ammonium tetrafluoroborate in a high yield. SOLUTION: This method for producing a quaternary ammonium tetrafluoroborate comprises reacting a quaternary ammonium bicarbonate or carbonate monoester salt of the general formula: Q+.RCO3- (R is hydrogen atom or a 1-4C alkyl; Q+ is a quaternary ammonium group) with hydrogen fluoride and subsequently reacting the obtained quaternary ammonium fluoride with boron trifluoride or a complex containing the boron trifluoride.